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54 Hydration process of nitrile compound.

57 Disclosed herein is a process for the hydration of a nitrile compound. Owing to the use of a Raney copper catalyst having an iron content of 0.7 wt. % or less based on the copper component in a suspended state, the process allows the catalyst to retain high activity and facilitates the separation of the catalyst and liquid reaction mixture by sedimentation and/or filtration. In an example, a catalyst with 0.1 % iron achieved 67 % conversion of acrylonitrile to acrylamide, whereas a catalyst with 1.56 % iron achieved only 40 %.

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HYDRATION PROCESS OF NITRILE COMPOUND

This invention relates to a process for hydrating a nitrile compound in the presence of a Raney copper catalyst in a liquid-phase suspended-bed to synthesize its corresponding acid amide compound, and specifically to an improved process for the hydration of a nitrile compound. It preferably uses a catalyst which retains high activity; and is advantageous where the catalyst is separated by sedimentation and/or filtration to recover the liquid reaction mixture.

Regarding the hydration of nitrile compounds in liquid phases and in the presence of Raney copper catalysts for the synthetic preparation of their corresponding acid amides, many proposals have heretofore been made including, for example, the preparation of nicotinamide (Japanese Patent Publication No. 22710/1973), the synthesis of acrylamide (Japanese Patent Publication No. 30810/1974), the effectiveness of addition of a copper salt selected from copper sulfate, copper nitrate, copper halides and the copper salts of fatty acids in an amount of 5 - 300 ppm based on water to a reaction system (Japanese Patent Publication No. 12409/1975),

the improved development of Raney copper alloys (Japanese Patent Publication No. 26910/1980), and the preparation of copper catalysts inclusive of Raney copper catalysts (U.S. Patent No. 3,767,706).

5 On the other hand, the following illustrative proposals have also been known on the effects of iron component in catalyst or reaction systems useful for the hydration reactions of nitrile compounds. Namely, Japanese Patent Laid-Open No. 13312/1975 discloses that
10 the service life of the catalyst can be prolonged by causing one or more of various nitrates inclusive of iron nitrate to exist in an amount of 1 - 600 ppm as nitrate radicals in a system useful for the hydration reaction of a nitrile compound to be effected in the
15 presence of a metallic copper-base catalyst such as Raney copper. Japanese Patent Laid-Open No. 115412/1976 discloses that metallic copper, which has been obtained by reducing a cuprous salt in the presence of a ferrous salt, is effective as a hydration
20 catalyst for acrylonitrile and methacrylonitrile. Further, it is also proposed in Japanese Patent Publication No. 33612/1977 that a multi-component catalyst formed by incorporating iron and other metal components in Raney copper has high activity than Raney
25 copper itself. The last-mentioned Japanese patent

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publication contains an Examples in which Raney copper having an iron content of about 3% was employed.

The present inventors have unexpectedly found that the following problems are also involved in
5 reaction systems containing the above-described Raney copper catalyst with or without the above-mentioned compounds including iron added thereto, and catalyst systems composed of Raney copper and iron and other metal components added thereto.

10 First of all, the activity of each Raney copper catalyst varies considerably depending on its supply source. Even those prepared in accordance with the same preparation process, their activities are different from one preparation lot to another. When a
15 powdery or granular Raney copper catalyst is used in a state suspended in a liquid reaction mixture, its readiness in sedimentation or filtration differs depending on its supply source or even its preparation lot upon separation of the catalyst from the liquid
20 reaction mixture by sedimentation or filtration.

Needless to say, it is important to solve the problem that the activities of catalysts vary depending on their supply sources as mentioned above. When such a catalyst was poor in the sedimentation and filtration
25 readiness in industrial production, unduly large sedimentation and filtration facilities were required.

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Furthermore, an economical loss such as unavoidable loss of the catalyst to an intolerably large amount due to its runoff and suspension of the operation due to clogging of filters or the like was also brought about.

5 Since the readiness in sedimentation and filtration varied from one preparation lot of catalyst to another, no stable operation was feasible.

Under the aforementioned circumstances, it has been desired to impart high reactivity and good
10 sedimentation and filtration readiness at the same levels to Raney copper catalysts.

The invention may enable one to provide a Raney copper catalyst capable of retaining high
15 activity for a long service time when causing a nitrile compound to react with water in the presence of a Raney copper catalyst to prepare its corresponding acid amide.

The invention may enable one to provide a
20 catalyst system featuring excellent sedimentation and filtration readiness upon effecting a reaction in the presence of a Raney copper catalyst in a suspended state and after the reaction, separating the catalyst and liquid reaction mixture from each other by
25 sedimentation and filtration.

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According to the invention, the process uses a Raney copper catalyst having an iron content of 0.7 wt.% based on the copper component in a process in which a nitrile compound is hydrated in the presence of a Raney copper catalyst while controlling the weight ratio of the nitrile compound to water at 60:45 - 5:95, the reaction temperature at 70 - 200°C and the conversion to the corresponding acid amide at 10 - 98%.

10 The above process may be effective for effecting a hydration reaction in the presence of a Raney copper catalyst in a suspended state and after the reaction, separating the catalyst and liquid reaction mixture from each other by sedimentation and/or filtration to recover the liquid reaction mixture. Embodiments may be particularly effective, for example, in separating the catalyst through a filter disposed within a reactor and drawing the liquid reaction mixture out of the reactor or in
15 separating the catalyst and liquid reaction mixture from each other by means of a thickener disposed within the reactor and then separating a small amount of the catalyst, which is still left in a state suspended in the liquid reaction mixture, by filtration either
20 inside or outside the reactor.

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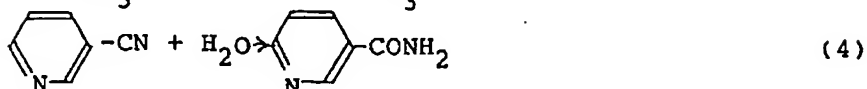
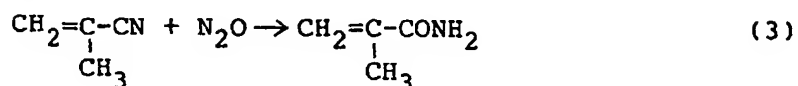
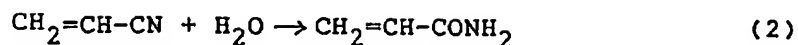
In the above process, the acid amide compound can be synthesized at a stable level of turnout by using a small amount of Raney copper catalyst. Moreover, the separation of the catalyst and liquid
5 reaction mixture by sedimentation and filtration can also be effected stably with ease. Accordingly, the above process has very large advantages for industrial production.

The present invention will hereinafter be
10 exemplified in detail.

Nitrile compounds useful as starting materials in the present invention may include, for example, monovalent aliphatic nitriles such as acetonitrile and propionitrile, multivalent aliphatic nitriles such as
15 malondinitrile, succinonitrile and adiponitrile, unsaturated aliphatic nitriles such as acrylonitrile and methacrylonitrile, aromatic nitriles such as benzonitrile and nicotinonitrile, and so on.

Reactions in which these nitrile compounds are
20 hydrated into their corresponding acid amide compounds are shown by the general formula (1). When acrylonitrile, methacrylonitrile and nicotinonitrile are subjected, by way of example, to hydration reactions respectively, acrylamide, methacrylamide and
25 nicotinoamide are prepared as illustrated in their corresponding chemical equations (2), (3) and (4).

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5 The Raney copper catalyst useful in the practice of the process of this invention is obtained by developing a so-called Raney copper alloy with an aqueous solution of a basic compound such as sodium hydroxide to leach out all soluble components from the

10 alloy and then washing and removing the majority of the leached-out compounds and any excess basic compound with water or the like. Hence, its principal catalytic component is believed to be metallic copper. As soluble component or components in the alloy, may be

15 mentioned aluminum and/or zinc. Of such soluble components, aluminum is suitable. The content of aluminum may preferably range from 30 to 70 wt.%. As the basic compound employed for the development of the alloy, it is possible to use inorganic compounds such

20 as sodium hydroxide, sodium carbonate and potassium hydroxide and organic amine compounds such as trimethylamine with sodium hydroxide being preferred.

The catalyst must be in the form of either powder or small granules so that it may be used in a

25 suspended-bed reactor. Catalysts having grain or

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particle sizes of 40 mesh or smaller as measured by the Tyler sieve system are generally used. In order to obtain these grain or particle sizes, it is necessary to develop powdery or granular Raney copper alloys
5 having such grain or particle sizes or to develop Raney alloys of larger grain or particle sizes followed by their grinding into smaller grains or particles.

In order to enhance its activity or selectivity, the catalyst may additionally contain a second
10 component such as nickel, molybdenum, silver or palladium. Where a nickel-containing Raney copper catalyst is desired by way of example, it may be prepared by developing a three-component Raney copper of aluminum-copper-nickel into a two-component Raney
15 catalyst of copper-nickel for use in the above reaction. Here, no problem or inconvenience will be encountered even if the resulting two-component Raney catalyst has a higher content of the second component than that of copper. For example, the ratio of nickel as
20 the second component to copper may be in the range between 55/45 and 85/15, and more typically it may be 70/30.

It is desired to keep the catalyst out of contact with oxygen or any oxygen-containing gas during
25 its preparation and storage. Oxygen may not impair the activity of the catalyst or may inversely enhance its

activity so long as the reaction of oxygen with the catalyst is controlled below a certain level. Beyond this level, its activity is however deleteriously affected and its sedimentation and filtration readiness
5 from the liquid reaction mixture is hence impaired. If a catalyst, which has in advance been brought into contact with oxygen, is used for the hydration reaction of acrylonitrile, another problem will arise that the more byproducts such as ethylene cyanhydrin will be
10 formed beside the intended acrylamide.

Synthesis of acrylamide through the hydration of acrylonitrile with such a Raney copper catalyst will next be illustrated by way of example.

A Raney copper catalyst is kept in a state
15 suspended in a liquid reaction mixture and is used in either continuous or batchwise system.

The weight ratio of acrylonitrile to water to be subjected to the hydration reaction may basically be optional. However, it may preferably be 60:40 - 5:95
20 or more preferably 50:50 - 10:90. The preferable reaction temperature for the hydration reaction may be within the range of 70 - 150°C or more specifically 90 - 140°C. The conversion of acrylonitrile to acrylamide may preferably be 10 - 98% or more
25 preferably 30 - 95%.

The above-described weight ratio of acrylonitrile to water, reaction temperature and conversion of acrylonitrile are applicable to general preparation conditions upon reacting nitrile compounds
5 and water in the presence of a Raney copper catalyst in suspended state to prepare their corresponding acid amides. These conditions are particularly effective when a Raney copper catalyst having an iron content of 0.7 wt.% or less based on the copper component is used.

10 If the iron content in a Raney copper catalyst to be used exceeds 0.7 wt.% based on the copper content, there will be brought about such inconvenience that as demonstrated by Examples to be given herein, the activity of the catalyst will be low to a
15 significant extent, and the sedimentation and filtration readiness of the catalyst upon separation of the catalyst from the liquid reaction mixture will decrease rapidly with time during the reaction run and as specific inconvenience, the
20 filtration resistance of a catalyst filter will increase. On the other hand, there is a practical limitation in minimizing the iron content in the catalyst. Therefore, the preferred iron content may range from 0.1 to 0.4 wt.%.

25 Even under the above-described preparation conditions, the three components of the unreacted

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acrylonitrile, unreacted water and resulting acrylamide may not form any homogeneous liquid system in some instances. To avoid this problem, synthesized acrylamide may be added back again to the reaction
5 system as a co-solvent or another inert co-solvent may also be used.

The interior of the reactor is kept at a pressure which is the sum of vapor pressures of the individual reaction components at the above-described
10 temperature or the total of sum of such vapor pressures and the pressure of an inert gas such as nitrogen gas. The interior pressure may normally be within the range of from normal pressure to 20 atmospheres.

Oxygen dissolved in the catalyst slurry,
15 acrylonitrile, water, co-solvent and the like, which are charged to the reactor, affects deleteriously on the activity of the catalyst, impairs the sedimentation and separation of the catalyst from the liquid reaction mixture and produces more byproducts such as
20 ethylenecyanhydrin. It is therefore desirable to remove such accompanying oxygen fully. For the same reasons, it is desirable to maintain the interior of the reactor as an oxygen-free atmosphere.

The liquid reaction mixture obtained in the
25 above reaction is then subjected to usual evaporation or distillation to obtain a concentrated aqueous

solution of acrylamide. At the same time, substantially all the unreacted acrylonitrile and a portion of water are distilled off for their recovery. Although the thus-recovered acrylonitrile and water may
5 be used in other application fields, they are generally used as the raw materials.

The hydration reaction of acrylonitrile has now been described in detail. The hydration reaction of methacrylonitrile is of the same notion and may hence
10 be carried out in much the same way. Although the hydration reaction of nicotinonitrile is substantially of the same notion, a reaction temperature higher than that employed for acrylonitrile or methacrylonitrile, for example, a temperature up to 200°C may be
15 employed.

The catalyst which has been used in the suspended state in the above-described hydration of the nitrile compound is usually separated immediately from the liquid reaction mixture. In some instances, it may
20 however be separated from a concentrated liquid reaction mixture which is to be obtained subsequent to the above-described evaporation or distillation. The separation may generally be carried out by sedimentation and/or filtration. As a specific manner
25 for effecting the sedimentation, it may be mentioned to use a thickener disposed within or outside the reactor

or to use a centrifugal thickener. On the other hand, as a specific manner for effecting the filtration, may be mentioned to employ a filter disposed either inside or outside the reactor or to use a centrifugal filter.

5 These sedimentation and filtration may also be combined.

The thus-separated catalyst may preferably be used repeatedly for the reaction. When a thickener is disposed within such a reactor in a continuous flow
10 system as disclosed, for example, in U.S. Patent No. 3,9985,806, the catalyst may be used continuously while holding the catalyst within the reactor as is.

When the reaction is effected in the above exemplified manner, the thus-separated liquid reaction
15 mixture may be drawn out of the reactor and a small amount of fine particles of the catalyst, which is still left in a state suspended in the liquid reaction mixture, may be removed almost completely by filtration. It may also be feasible to dispose a
20 filter within a reactor to remove the catalyst completely inclusive of its fines suspended in the liquid reaction mixture. These systems of thickener and filter, in combination or not, may be chosen as desired.

25 As mentioned above, the present invention makes use of a Raney copper catalyst having a particularly

low iron content. As principal sources for the iron which is mixed in the Raney copper catalyst, may be contemplated (1) iron contained in aluminum, zinc and/or the like which are employed as soluble
5 components in a Raney alloy, (2) iron mixed in as a result of wearing or corrosion of steel-made facilities employed upon grinding and classifying a Raney alloy to a desired range of particle sizes, (3) iron contained in sodium hydroxide and water which are both employed
10 for the development of a Raney alloy, (4) iron mixed in as a result of wearing or corrosion of steel-made developing facilities.

In order to avoid the activity reduction of the catalyst and the reduction of its sedimentation and
15 filtration readiness due to these various causes, it is necessary to implement various countermeasures so that the incorporation of iron by the above-mentioned causes can be minimized effectively.

As specific examples of the above-mentioned
20 countermeasures, it may be mentioned (1) to use the limited brands or grades of aluminum or zinc to prepare the alloy, for example, to avoid use of aluminum or zinc of such a high iron content as 0.4 wt.%; (2) to
use grinding and classification facilities made of
25 stainless steel instead of carbon steel; (3) to use sodium hydroxide having an iron content as low as

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possible and to avoid use of storage and transportation equipment made of carbon steel; (4) to use soft water, deionized water or the like as developing water and to avoid use of storage and transportation equipment made of carbon steel; (5) to use developing facilities made of stainless steel; etc.

By achieving the total control in such manners as mentioned above, it has become feasible for the first time to use a Raney catalyst having an iron content of 0.7 wt.% or less. Owing to the use of such a Raney catalyst, the above-described objects and effects have been fulfilled. Of the above-mentioned various countermeasures, the control of iron content in aluminum and the selection of material for the grinding and classification facilities are particularly important. Lack of these countermeasures appears to result in a great deal of iron left in a developed Raney copper.

The term "reduced sedimentation readiness of the catalyst used in this invention" as used herein means, for example, that when stirring is stopped in the course of use of an agitator-equipped reactor, the suspended particles or grains of the Raney copper catalyst become more difficult in sedimentation. When the reaction is effected for example in a reactor provided with a partition wall to form a catalyst

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sedimentation region (i.e., thickener), where the
turbulence is not applied to the catalyst slurry, and
the precipitated catalyst is recycled to the
catalyst-suspended region and the catalyst-separated
5 liquid reaction mixture is drawn out of the reactor,
the above term "reduced sedimentation readiness of the
catalyst used in this invention" means that the
thus-drawn liquid reaction mixture contains more
catalyst which is still remaining in a suspended state
10 without undergoing sedimentation. Increase of such
suspended catalyst means a direct loss in the effective
amount of the catalyst, leading to an increase of the
catalyst consumption.

On the other hand, the term "reduced filtration
15 readiness" as used herein means that the filtration
resistance has been increased in a catalyst filter
provided within or outside a reactor as will be
demonstrated in Examples herein. Such a phenomenon
will lead to a reduction of the production capacity.

20 This invention will next be described by the
following Examples:

Example 1:

A 1:1 alloy of copper and aluminum was ground
and sifted to obtain Raney copper alloy powder having
25 particle size less than 80 mesh. The alloy powder was
developed with caustic soda in a usual manner, followed

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by its thorough washing with water to prepare a Raney copper catalyst. From a number of catalyst lots obtained in the above manner, those having iron contents of 0.10, 0.24, 0.41, 0.70, 1.15 and 1.56%,
5 based on their corresponding copper components, were used. The analysis of iron contents was effected by atomic absorption spectroscopy subsequent to dissolution of the catalysts in nitric acid.

Incidentally, the iron contents of the
10 thus-chosen Raney alloys of 6 different lots before grinding and sifting and the material of a grinding mill employed are given in Table 1.

Using these six catalysts of different lots, the following batchwise reaction tests were conducted.
15 Charged in a 100-ml flask equipped with a stirrer, thermometer and reflux condenser were 6.6 g of acrylonitrile, 36 g of water and 7 g of one of the catalysts. Since the catalyst was prepared and stored in a state immersed in water, about 7 g of water was
20 accompanied when the catalyst was charged. Thus, this amount had in advance been deducted from the amount of water to be charged separately so that the total amount of water was set at 36 g.

The flask was heated externally over a bath to
25 about 70°C. Thereafter, the contents were reacted for 2 hours while adjusting the temperature of the bath to

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maintain the internal temperature of the flask at 70 -
73°C.

Any dissolved oxygen had in advance been removed
from the raw materials. In the subsequent operation,
5 the penetration of air was avoided so as to prevent the
catalyst from being brought into contact with oxygen.
Two hours later, the flask was cooled to terminate the
reaction and the liquid reaction mixture was analyzed
by gas chromatography to determine the conversion of
10 acrylonitrile to acrylamide. Results are summarized in
Table 1.

Table 1

Run No.	Iron content in alloy before grinding (wt.%; based on alloy)	Material of grinding mill	Iron content (%) based on copper	Conversion (%)	Classification
1-1	0.04	stainless steel	0.10	67	Example
1-2	0.11	ditto	0.24	65	ditto
1-3	0.18	ditto	0.41	63	ditto
1-4	0.11	carbon steel	0.70	60	ditto
1-5	0.33	ditto	1.15	49	Comp. Example
1-6	0.57	ditto	1.56	40	ditto

Note: Stainless steel - SUS 304 was used.

Carbon steel - SS 41 was used.

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When a Raney copper is used for the industrial production of acrylamide, its conversion is preferably 60% or higher in the above-described activity evaluation test. It is envisaged from Table 1 that its iron content must be about 0.7% or less to achieve such a preferable conversion.

Example 2:

Using the three catalysts of Example 1 which had iron contents of 0.24, 0.70 and 1.56% respectively, the following continuous flow reaction tests were conducted.

One hundred fifty parts of one of the catalysts were charged in a stainless steel reactor equipped with a stirrer and a built-in catalyst filter, to which acrylonitrile and water from both of which any dissolved oxygen had in advance been removed by blowing nitrogen gas thereinto were charged at flow rates of 300 and 700 parts by weight per hour respectively. The reaction was carried out at 120°C while stirring the reaction system to maintain the catalyst in a suspended state. As a reaction promoter, copper nitrate was added to the water to be fed in such an amount that its concentration reached 20 ppm of the liquid reaction mixture as measured in terms of nitrate radicals.

The liquid reaction mixture was caused to flow through the catalyst filter and was then taken out of

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the reactor as a liquid substantially free of the catalyst. It thereafter flowed into a reaction liquid reservoir of a hermetically-sealed structure. It was drawn out of the reservoir once a day.

5 The liquid reaction mixture, which flowed out of the reactor, was sampled at intervals of two days and analyzed by gas chromatography to determine the conversion of acrylonitrile to acrylamide.

10 The pressure of the reaction liquid reservoir was always maintained at $4 \text{ Kg/cm}^2(\text{G})$ by either charging or discharging nitrogen gas. In this manner, the reactor has a pressure somewhat higher than 4 Kg/cm^2 . This pressure difference (ΔP) indicates primarily the resistance of the filtration area of the catalyst filter.

15 ΔP was close to 0 on the first day of the reaction. It began to increase as the reaction was continued. The reaction was stopped when ΔP exceeded 2 Kg/cm^2 .

20 The above reaction test was conducted by using the above-described three catalysts of different types separately. Results are given in Table 2. Namely, higher conversions and smaller ΔP were maintained during the test period of 14 days when the catalyst
25 having the lower iron content was employed. However, with the catalyst having the higher iron content, the

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conversion remained low and the ΔP increased in an earlier stage of the test, resulting in termination of the test.

Table 2

Run No.	2-1		2-2		2-3	
Fe content(%; on Cu)	0.24		0.70		1.56	
Evaluated Item	Conversion (%)	ΔP (Kg/cm)	Conversion (%)	ΔP (Kg/cm)	Conversion (%)	ΔP (Kg/cm)
<u>Days passed during the reaction test</u>						
0	72	0	68	0	51	0
2	67	0	63	0	46	0
4	64	0	60	0	42	0.4
6	60	0	56	0	39	1.1.
8	57	0.1	54	0.2	Test was stopped after 7 th day.	
10	54	0.2	52	0.2		
12	52	0.2	49	0.4		
14	50	0.3	48	0.6		
Classification	Example		Example		Comparative Example	

Example 3:

Using the same 6 catalysts of different lots as those employed in Example 1, reaction tests were conducted in the same manner and under the same conditions as in Example 1 except that acrylonitrile was replaced by methacrylonitrile. Results are shown in Table 3.

Table 3

Run No.	Fe-content (%) based on Cu	Conversion (%)	Classification
3-1	0.10	54	Example
3-2	0.24	53	ditto
3-3	0.41	51	ditto
3-4	0.70	49	ditto
3-5	1.15	39	Comp. Example
3-6	1.56	33	ditto

Example 4:

10 Using the same 3 catalysts of different lots as those employed in Example 2, continuous flow reaction tests were conducted in the same manner and under the same conditions as in Example 2 except that acrylonitrile was replaced by methacrylonitrile.

15 Results are shown in Table 4.

Table 4

Run No.	4-1		4-2		4-3	
Fe content(%; on Cu)	0.24		0.70		1.56	
Evaluated Item	Conversion (%)	ΔP (Kg/cm)	Conversion (%)	ΔP (Kg/cm)	Conversion (%)	ΔP (Kg/cm)
<u>Days passed during the reaction test</u>						
0	61	0	58	0	45	0
2	57	0	54	0	43	0
4	55	0	51	0	42	0.2
6	53	0	49	0	40	0.8
8	50	0	47	0	38	1.7
10	47	0	44	0.2	Test was stopped after 8 th day.	
12	45	0.1	42	0.3		
14	44	0.1	42	0.3		
Classification	Example		Example		Comparative Example	

Example 5:

Using the same 6 catalysts of different lots as those employed in Example 1, reaction tests were conducted in the same manner and under the same conditions as in Example 1 except that acrylonitrile was replaced by nicotinonitrile. Results are shown in Table 5.

Table 5

Run No.	Fe-content (%; on Cu)	Conversion (%)	Classification
5-1	0.10	75	Example
5-2	0.24	74	ditto
5-3	0.41	71	ditto
5-4	0.70	68	ditto
5-5	1.15	56	Comp. Example
5-6	1.56	52	ditto

Claims:

1. A process for hydrating a nitrile compound
in the presence of a Raney copper catalyst while
controlling the weight ratio of the nitrile compound to
5 water at 60:40 - 5:95, the reaction temperature at 70 -
200°C and the conversion of the nitrile compound to
the corresponding acid amide at 10 - 98%, which
comprises using, as the catalyst, a Raney copper
catalyst having an iron content of 0.7 wt.% or less
10 based on the copper component thereof.

2. A process as claimed in Claim 1, wherein a
Raney copper catalyst having an iron content of 0.1 -
0.4 wt.% based on the copper component is used.

15 3. A process as claimed in claim 1 or 2, wherein the
hydration reaction is carried out while maintaining the
Raney copper catalyst in a suspended state, and after
the reaction, the catalyst and the liquid reaction
20 mixture are separated from each other by sedimentation
and/or filtration so as to recover the liquid reaction
mixture.

4. A process as claimed in Claim 3, wherein the
catalyst and liquid reaction mixture are separated from
25 each other by means of a filter disposed within a
reactor.

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5. A process as claimed in Claim 3, wherein the catalyst and liquid reaction mixture are separated from each other by means of a thickener disposed within a reactor, and a small amount of the catalyst which is
5 still left in a state suspended in the liquid reaction mixture is then separated by means of a filter disposed within the reactor.

6. A process as claimed in Claim 3, wherein the
10 catalyst and liquid reaction mixture are separated from each other by means of a thickener disposed within a reactor, and a small amount of the catalyst which is still left in a state suspended in the liquid reaction mixture is then separated by means of a filter provided
15 outside the reactor.

7. A process as claimed in any preceding claim wherein the nitrile compound is acrylonitrile.

8. A process as claimed in any preceding claim
20 wherein the nitrile compound is methacrylonitrile.

9. A process as claimed in any preceding claim wherein the nitrile compound is nicotinonitrile.

10. A process according to any preceding claim
25 wherein the nitrile compound to water ratio is 60:45 - 5:95.